# Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders:

**P-07-0087;**

**P-08-0200;**

**P-08-0643, P-08-0642, P-08-0644, and P-08-0664;**

**P-08-0748 and P-08-0751;**

**P-09-0245 and P-09-0246;**

**P-09-0293 and P-09-0294;**

**P-10-0058, P-10-0059, and P-10-0060;**

**P-10-0148;**

**P-11-0091, P-11-0092, and P-11-0093;**

**P-12-0450**

**Prepared by:**

**October 2016**

**Report P-10-16-01**

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## Objective

The subject consent orders require the Company to analyze representative samples of the initially isolated formulations of the PMN substances at each manufacturing facility for the analytes specified in Table 3 of the consent orders and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulations and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulations of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent orders and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent orders. This report describes the analytical methods that are used for the required annual analytical reporting.

## Materials and Methods

### A. Samples Analyzed

1.

Until January of 2016 Lots of starting material are manufactured at shipped to for and then returned to or other facilities for conversion into the PMN substance. Each lot of is analyzed at to ensure the starting material is in compliance with and does not exceed the limits specified

in Tables 1 and 2 of the consent orders. Additionally, will analyze some lots using a more sensitive analytical method. Starting in January of 2016 lots of starting material are manufactured and thermally treated at and then used at or shipped to other facilities for conversion into the PMN substance.

2. Initially Isolated Formulations of the PMN Substance

Representative samples of the Initially Isolated Formulations of the PMN substance manufactured or imported by the Company at each manufacturing facility are analyzed at initial commencement of manufacture, and at least annually thereafter. In addition, if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered, the initially isolated formulation of the PMN substance is analyzed again and reported to EPA. The initially isolated formulations of the PMN substance are analyzed for the analytes specified in Table 3 of the consent orders and reported to EPA.

**B. Analytical Methods**

1. Analytical Method for

Until January of 2016 determination of residual of the determined by GC-FID at by using normalized area percent. Some lots of also get analyzed by GC-

FID at but these additional analyses are not required by consent order. The methods are discussed in detail in Appendices 1 and 2. Starting in January of 2016 determination of residual of the is determined by GC-FID at A GC-FID method is sufficient for quantitative analysis of impurities in demonstrate compliance with the limits specified in Table 1 of the consent orders; however, a GC-MS method is used on some lots of at for the determination of residual because some values fall below the limit of detection of the GC-FID method. Either method is adequate for determining compliance with the limits specified in Table 1 of the consent orders. The limit of quantitation for all analytes specified in Table 1 of the consent orders by GC-FID at and s 100 ppm, and the limit of quantitation for all analytes by GC-MS is 2 ppm. The GC-MS method is discussed in detail in Appendix 3.

Until January of 2016 for determining compliance with the limits specified in Table 2 of the consent orders, the determination at is achieved by GC-MS analysis of the The limit of quantitation of this method is 2 ppm. This method is discussed in detail in Appendix 4. Starting in January of 2016 for determining compliance with the limits specified in Table 2 of the consent orders, the determination at is achieved by GC-MS analysis of the The limit of quantitation of this method is 2 ppm.

At is quantified in some lots of the by using a modified method based on the published method by

Samples are prepared and analyzed with a fortified sample for spike recovery determination. The limit of quantitation for is 2 ppm or 0.5 ppm as is dependent on the sample prep. This method is discussed in detail in Appendix 3.

## Results and Discussion

## A. Analysis of

A summary of the results, including the range and averages, for analytes specified in Table 1 and Table 2 of the consent orders are presented in specific reports for the starting material.

## B. Analysis of the Initially Isolated Formulations of the PMN Substances

The results for analytes specified in Table 3 of the consent orders are presented in specific reports for each consent order.

## Conclusion

The analytical methods for the analyses specified in the consent orders for the starting material and all initially isolated formulations of the PMN substances have been presented in this report and will be referenced in all future analytical reports submitted to EPA.

# Appendix 1

**Analysis of Under Consent Orders:**

**P-07-0087;**

**P-08-0200;**

**P-08-0643, P-08-0642, P-08-0644, and P-08-0664;**

**P-08-0748 and P-08-0751;**

**P-09-0245 and P-09-0246;**

**P-09-0293 and P-09-0294;**

**P-10-0058, P-10-0059, and P-10-0060;**

**P-11-0091, P-11-0092, and P-11-0093**

# Annual Report

August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-02**

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## Objective

The subject consent orders require the Company to analyze representative samples of the initially isolated formulations of the PMN substances at each manufacturing facility for the analytes specified in Table 3 of the consent orders and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulations and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulations of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent orders and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent orders. This report covers these annual reporting requirements for the starting material. The initially isolated formulations of the PMN substance results are provided in separate reports for their respective consent orders.

## Materials and Methods

### A. Samples Analyzed

A total of thirty three (33) lots of were prepared between August 11, 2015 and August 10, 2016 and designated as Lots 166 through 174 and Lots 1 through 26. Lots 166 through 174 was manufactured at

shipped to for and then returned to or shipped to other facilities for conversion into the PMN substances. Starting in January of 2016 Lots 1 through 26 were manufactured and thermally treated at and then shipped to other facilities for conversion into the PMN substances. In this reporting period, was analyzed at or depending on thermal treatment location, to ensure successful As required in the consent orders, the has been analyzed for the analytes shown in Tables 1 and 2 of the consent orders.

**B. Analytical Methods**

Analytical Method for

Determination of residual

of the is determined by GC-FID at by using normalized area percent. Some lots of also get analyzed by GC/MS a but these additional analyses are not required by consent order. Starting in January 2016 the determination of residual

of the is determined by GC-FID at by using normalized area percent. determination at is achieved by GC-MS analysis of th The above methods are discussed detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Results

### Analysis of

A summary of the results, including the range, averages, and outlying data, for analytes specified in Table 1 of the consent orders are presented in Table 1-A (GC-FID; and 1-B (GC/MS;

| LOT # | Method | *conc (%)* | *conc (%)* | *conc (%)* | *conc (%)* | *conc (%)* | *conc (%)* |
|---|---|---|---|---|---|---|---|
| Limit | | 89% min | 6% max | 1.4 max | 0.1% max | 1.4% max | 0.1% max |
| Limit for Consent Order P-10-0148 | | 93% min | 4% max | 0.4% max | 0.1% max | 0.4% max | 0.1% max |
| 166 | GC-FID | 97.71 | 0.03 | 0.05 | <0.01 | <0.01 | 0.02 |
| 167 | GC-FID | 97.82 | 0.02 | 0.07 | <0.01 | <0.01 | 0.01 |
| 168 | GC-FID | 97.48 | 0.13 | 0.08 | <0.01 | <0.01 | 0.01 |
| 169 | GC-FID | 97.98 | 0.03 | 0.06 | <0.01 | <0.01 | 0.01 |
| 170 | GC-FID | 97.91 | <0.01 | 0.05 | <0.01 | <0.01 | <0.01 |
| 171 | GC-FID | 97.99 | <0.01 | 0.08 | <0.01 | <0.01 | 0.01 |
| 173 | GC-FID | 97.98 | 0.02 | 0.06 | <0.01 | <0.01 | 0.02 |
| 174 | GC-FID | 97.25 | 0.03 | 0.07 | <0.01 | <0.01 | 0.02 |
| 1 | GC-FID | 98.8 | 0.013 | <0.01 | <0.01 | 0.07 | <0.01 |
| 2 | GC-FID | 99 | 0.012 | <0.01 | <0.01 | <0.01 | <0.01 |
| 3 | GC-FID | 98.63 | <0.01 | <0.01 | <0.01 | 0.12 | <0.01 |
| 4 | GC-FID | 97.89 | <0.01 | 0.293 | <0.01 | 0.068 | <0.01 |
| 5 | GC-FID | 98.531 | 0.016 | 0.075 | 0.007 | 0.089 | <0.004 |
| 6 | GC-FID | 97.45 | 0.022 | 0.21 | 0.006 | 0.651 | <0.004 |
| 7 | GC-FID | 97.736 | 0.019 | 0.089 | 0.004 | 0.565 | <0.004 |
| 8 | GC-FID | 96.807 | 0.009 | 0.092 | 0.01 | 0.984 | <0.004 |
| 9 | GC-FID | 97.696 | 0.013 | 0.173 | 0.006 | 0.056 | <0.004 |
| 10 | GC-FID | 97.155 | 0.02 | 0.121 | 0.01 | 0.557 | <0.004 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 11 | GC-FID | 97.685 | 0.018 | 0.061 | 0.022 | 0.346 | <0.004 |
| 12 | GC-FID | 97.246 | 0.028 | 0.226 | 0.012 | 0.517 | <0.004 |
| 13 | GC-FID | 97.409 | 0.023 | 0.053 | 0.011 | 0.562 | <0.004 |
| 14 | GC-FID | 97.47 | 0.018 | 0.041 | 0.009 | 0.528 | <0.004 |
| 15 | GC-FID | 97.86 | 0.024 | 0.09 | 0.01 | 0.172 | <0.004 |
| 16 | GC-FID | 98.22 | 0.031 | 0.154 | 0.058 | 0.147 | <0.004 |
| 17 | GC-FID | 97.75 | 0.025 | 0.102 | 0.03 | 0.351 | <0.004 |
| 18 | GC-FID | 97.794 | 0.012 | 0.372 | 0.023 | 0.164 | <0.004 |
| 19 | GC-FID | 97.807 | 0.009 | 0.111 | 0.012 | 0.264 | <0.004 |
| 20 | GC-FID | 97.284 | 0.016 | 0.321 | 0.01 | 0.077 | <0.004 |
| 21 | GC-FID | 97.77 | 0.02 | 0.193 | 0.011 | 0.2 | <0.004 |
| 22 | GC-FID | 97.658 | 0.013 | 0.06 | 0.043 | 0.424 | <0.004 |
| 23 | GC-FID | 97.456 | 0.008 | 0.247 | 0.016 | 0.271 | <0.004 |
| 24 | GC-FID | 97.197 | 0.022 | 0.211 | 0.011 | 0.242 | <0.004 |
| 25 | GC-FID | 97.897 | 0.029 | 0.238 | 0.014 | 0.12 | <0.004 |
| 26 | GC-FID | 97.719 | 0.016 | 0.194 | 0.019 | 0.155 | <0.004 |
| Avg | | 97.77 | 0.02 | 0.13 | 0.01 | 0.23 | <0.01 |
| Range | | 96.81 -- 99.00 | <0.01 -- 0.13 | <0.01 -- 0.372 | <0.01 -- 0.058 | <0.01 -- 0.984 | <0.004 -- 0.02 |

**Table 1-A**

| LOT # | Method | *conc (%)* | *conc (%)* | *conc (%)* | *conc (%)* |
|---|---|---|---|---|---|
| Limit | | 6% max | 1.4 max | 0.1% max | 0.1% max |
| 166 | GC/M | 0.0175 | 0.0260 | <0.0002 | <0.0002 |
| 5 | GC/M | 0.0267 | 0.0209 | <0.00002 | <0.0002 |
| 10 | GC/M | 0.0321 | 0.0396 | <0.0002 | 0.0006 |
| 15 | GC/M | 0.0379 | 0.0277 | <0.0002 | 0.0004 |
| 20 | GC/M | 0.0251 | 0.0908 | <0.0002 | 0.0003 |
| 25 | GC/M | 0.0517 | 0.065 | <0.0002 | 0.0005 |
| Avg | | 0.03 | 0.045 | <0.0002 | 0.0003 |
| Range | | 0.0175 -- 0.0517 | 0.0209 -- 0.0908 | <0.0002 -- <0.0002 | <0.0002 -- 0.0006 |

**Table 2-B**

The results for the analyte specified in Table 2 of the consent orders are presented in Table 2

| LOT # | *conc (PPM)* |
|---|---|
| Limit | 2 ppm max |
| 166 | <2 |
| 167 | <2 |
| 168 | <2 |
| 169 | <2 |
| 170 | <2 |
| 171 | <2 |
| 173 | <2 |
| 174 | <2 |
| 1 | <2 |
| 2 | <2 |
| 3 | <2 |
| 4 | <2 |
| 5 | <2 |
| 6 | <2 |
| 7 | <2 |
| 8 | <2 |
| 9 | <2 |
| 10 | <2 |
| 11 | <2 |
| 12 | <2 |
| 13 | <2 |
| 14 | <2 |
| 15 | <2 |
| 16 | <2 |
| 17 | <2 |
| 18 | <2 |
| 19 | <2 |
| 20 | <2 |
| 21 | <2 |
| 22 | <2 |
| 23 | <2 |
| 24 | <2 |
| 25 | <2 |
| 26 | <2 |
| Avg | <2 |
| Range | <2 -- <2 |

**Table 3**

## Conclusion

Per the subject consent orders, the starting material manufactured and used to produce PMN substance between August 11, 2015 and August 10, 2016 was analyzed and reported for: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1-A and 1-B of the consent orders and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent orders.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-07-0087

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-03**

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## Objective

Consent order P-07-0087 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substance only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

Materials and Methods

## A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substance covered under P-07-0087, there has been one (1) initially isolated formulation of the PMN substance manufactured. It is as follows:

The initially isolated formulation of the PMN substance listed above was packed out between August 11, 2015 and August 10, 2016. As per the consent order P-07-0087, representative samples of this formulation was analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

## B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substance were analyzed for the analytes specified in Table 3 of the P-07-0087 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of

and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulation of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Table 1, below.

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 60J | | 0.0004 | <0.0002 | <0.5 |
| Avg | | 0.0004 | <0.0002 | <0.5 |
| Range | | <0.0002 -- 0.0004 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1**

## Conclusion

The analyses specified in the consent order P-07-0087 have been completed for representative samples of the initially isolated formulation of the PMN substance manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-08-0200

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by**

**October 2016**

**Report P-10-16-04**

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## Objective

Consent Order P-08-0200 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substance only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Intially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substance covered under P-08-0200, there have been two (2) initially isolated formulations of the PMN substance manufactured. They are as follows:

The initially isolated formulations of the PMN substance listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-08-0200, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

### B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substance were analyzed for the analytes specified in Table 3 of the P-08-0200 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of

and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748

and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Tables 1A – 1B, below.

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 75J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- 0.0007 | <0.5 -- <0.5 |

**Table 1-A**

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 61J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-B**

## Conclusion

The analyses specified in the consent order P-08-0200 have been completed for representative samples of the initially isolated formulations of the PMN substance manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-08-0643, P-08-0642, P-08-0644, and P-08-0664

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-05**

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## Objective

Consent order P-08-0643, P-08-0642, P-08-0644, and P-08-0664 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substances only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been one (1) initially isolated formulation of the P-08-0642 substance, three (3) initially isolated formulations of the P-08-0643 substance, and one (1) initially isolated formulation of the P-08-0664 PMN substance manufactured. They are as follows:

The initially isolated formulations of the PMN substances listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-08-0643, P-08-0642, P-08-0644, and P-08-0664, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

## B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substances were analyzed for the analytes specified in Table 3 of the P-08-0643, P-08-0642, P-08-0644, and P-08-0664 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

# Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Tables 1A – 1D, below.

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 0033964000 | | <0.0002 | <0.0002 | <0.1 |
| 0035126400 | | <0.0002 | <0.0002 | <0.1 |
| 0041321000 | | <0.0002 | <0.0002 | <0.1 |
| Avg | | <0.0002 | <0.0002 | <0.1 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.1 -- <0.1 |

*LOQ = 0.1 ppm from alternate method described in report P-10-16-01

**Table 1-A**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 0040689800 | | <0.0002 | <0.0002 | <0.1 |
| 0039122300 | | <0.0002 | <0.0002 | <0.1 |
| Avg | | <0.0002 | <0.0002 | <0.1 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.1 |

*LOQ = 0.1 ppm from alternate method described in report P-10-16-01

**Table 1-B**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| S06.16 | | <0.0002 | <0.0002 | <0.5 |
| S37.16J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | 0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- 0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-C**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 20J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-D**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 0038120100 | | <0.0002 | <0.0002 | <0.1 |
| 0040382000 | | <0.0002 | <0.0002 | <0.1 |
| 0040871400 | | <0.0002 | <0.0002 | <0.1 |
| Avg | | <0.0002 | <0.0002 | <0.1 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.1 -- <0.1 |

**Table 1-E**

## Conclusion

The analyses specified in the consent order P-08-0643, P-08-0642, P-08-0644, and P-08-0664 have been completed for representative samples of the initially isolated formulations of the PMN substances manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-08-0748 and P-08-0751

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-06**

# Table of Contents

## Objective

Consent order P-08-0748 and P-08-0751 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substances only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been two (2) initially isolated formulations of the P-08-0751 PMN substance manufactured. They are as follows:

The initially isolated formulations of PMN substance listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-08-748 and P-08-751, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

### B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substance were analyzed for the analytes specified in Table 3 of the P-08-0748 and P-08-0751 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under

Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Tables 1A – 1C, below:

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| L85.15 | | 0.0004 | <0.0002 | <0.5 |
| Avg | | 0.0004 | <0.0002 | <0.5 |
| Range | | 0.0004 -- 0.0004 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-A**

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 20 | | <0.0002 | <0.0002 | <0.5 |
| L08.16 | | <0.0002 | <0.0002 | <0.5 |
| L46.16J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-B**

## Conclusion

The analyses specified in the consent order P-08-0748 and P-08-0751 have been completed for representative samples of the initially isolated formulations of the PMN substance manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-09-0245 and P-09-0246

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-07**

# Table of Contents

## Objective

Consent order P-09-0245 and P-09-0246 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substances only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been three (3) initially isolated formulations of the P-09-0245 PMN substance and one (1) initially isolated formulations of the P-09-0246 PMN substance manufactured. They are as follows:

The initially isolated formulations of PMN substances listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-09-0245 and P-09-0246, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

## B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substances were analyzed for the analytes specified in Table 3 of the P-09-0245 and P-09-0246 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

# Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Tables 1A – 1B, below:

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 222T | | <0.0002 | <0.0002 | <0.5 |
| 240 | | <0.0002 | <0.0002 | <0.5 |
| 242J | | 0.0007 | <0.0002 | <0.5 |
| 260J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | 0.0003 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0007 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-A**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 70J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-B**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 34J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-C**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 12 | | 0.0005 | 0.0012 | <0.5 |
| Avg | | 0.0005 | 0.0012 | <0.5 |
| Range | | 0.0005 -- 0.0005 | 0.0012 -- 0.0012 | <0.5 -- <0.5 |

**Table 1-D**

## Conclusion

The analyses specified in the consent order P-09-0245 and P-09-0246 have been completed for representative samples of the initially isolated formulations of the PMN substances manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-09-0293 and P-09-0294

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-08**

# Table of Contents

## Objective

Consent order P-09-0293 and P-09-0294 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substances only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been one (1) initially isolated formulation of the P-09-0294 and one (1) initially isolated formulation of the P-09-0293 PMN substance manufactured. They are as follows:

The initially isolated formulations of PMN substances listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-09-0294, the formulation was analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

### B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

The initially isolated formulations of the PMN substances were analyzed for the analytes specified in Table 3 of the P-09-0294 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-

09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Tables 1A – 1B, below:

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 10J | | 0.0003 | <0.0002 | <0.5 |
| Avg | | 0.0003 | <0.0002 | <0.5 |
| Range | | 0.0003 -- 0.0003 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-A**

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 8J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-B**

## Conclusion

The analysis specified in the consent order P-09-294 has been completed for the initially isolated formulations of the PMN substances manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze the initially isolated formulation(s) and

report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-10-0058, P-10-0059, and P-10-0060

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-09**

# Table of Contents

## Objective

Consent order P-10-0058, P-10-0059, P-10-0060 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for th starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substance only. Th starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis o Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been four (4) initially isolated formulations of the P-10-0060 substance manufactured. They are as follows:

The initially isolated formulations of the PMN substance listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-10-0060, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

### B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substance were analyzed for the analytes specified in Table 3 of the P-10-0060 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of

and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Tables 1A – 1D, below.

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 1058 | | <0.0002 | <0.0002 | <0.5 |
| 1064J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 |

**Table 1-A**

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 1071J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-B**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 1066J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-C**

| LOT # | Product | conc (%) | conc (%) | conc (ppm) |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 1077J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-D**

## Conclusion

The analyses specified in the consent order P-10-0058, P-10-0059, P-10-0060 have been completed for representative samples of the initially isolated formulations of the PMN substance manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-11-0091, P-11-0092, and P-11-0093

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by**

**October 2016**

**Report P-10-16-10**

# Table of Contents

## Objective

Consent order P-11-0091, P-11-0092, P-11-0093 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for th starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substance only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been one (1) initially isolated formulations of the P-11-0091 substance manufactured. They are as follows:

The initially isolated formulations of the PMN substance listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-11-0091, P-11-0092, P-11-0093, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

### B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substance were analyzed for the analytes specified in Table 3 of the P-11-0091, P-11-0092, P-11-0093 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and

P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Table 1A below.

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| S10.16 | | <0.0002 | <0.0002 | <0.5 |
| S62.16J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 -- <0.5 |

**Table 1-A**

## Conclusion

The analyses specified in the consent order P-11-0091, P-11-0092, P-11-0093 have been completed for representative samples of the initially isolated formulations of the PMN substance manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.

# Analysis of Initially Isolated Formulations of PMN Substance Required Under Consent Order PMN P-12-0450

## Annual Report

## August 11, 2015 – August 10, 2016 Analytical Summary

**Prepared by:**

**October 2016**

**Report P-10-16-11**

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## Objective

Consent order P-12-0450 requires the Company to analyze representative samples of the initially isolated formulation(s) of the PMN substance at each manufacturing facility for the analytes specified in Table 3 of the consent order and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulation(s) of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent order and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent order. This report covers the annual reporting requirements for the initially isolated formulations of the PMN substance only. The starting material results are provided in a separate report because the starting material is common to PMN substances manufactured under several different consent orders. The starting material results can be found in Report P-10-16-02 entitled, "Analysis of Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748 and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, P-11-0093, and P-12-0450"

## Materials and Methods

### A. Samples Analyzed

Initially Isolated Formulations of the PMN Substance

In this reporting period for the manufacture of the PMN substances covered under the subject consent order, there have been one (1) initially isolated formulations of the P-12-0450 substance manufactured. They are as follows:

The initially isolated formulations of the PMN substance listed above were packed out between August 11, 2015 and August 10, 2016. As per the consent order P-12-0450, representative samples of these formulations were analyzed for the substances specified in Table 3 of the consent order and reported to EPA at the time of first commercial manufacture or import, and again if any new manufacturing facility was added or if the process of manufacture of the PMN substance or any intermediate thereof was significantly altered, or at least annually in this report.

### B. Analytical Methods

Analytical Methods for Initially Isolated Formulations of the PMN Substance

Representative samples of the initially isolated formulations of the PMN substance were analyzed for the analytes specified in Table 3 of the P-12-0450 consent order using methods described in detail in Report P-10-16-01 entitled: "Analytical Methods for the Analysis of

and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders: P-07-0087; P-08-0200; P-08-0643, P-08-0642, P-08-0644, and P-08-0664; P-08-0748

and P-08-0751; P-09-0245 and P-09-0246; P-09-0293 and P-09-0294; P-10-0058, P-10-0059, and P-10-0060; P-10-0148; P-11-0091, P-11-0092, and P-11-0093."

## Results

### Analysis of Initially Isolated Formulations of the PMN Substance

The results for analytes specified in Table 3 of the consent order are presented in Table 1A below.

| LOT # | Product | *conc (%)* | *conc (%)* | *conc (ppm)* |
|---|---|---|---|---|
| | (estimated Max) | 0.7% max combined | 0.1% max combined | 2 ppm max |
| 2 | | <0.0002 | <0.0002 | <0.5 |
| 3J | | <0.0002 | <0.0002 | <0.5 |
| Avg | | <0.0002 | <0.0002 | <0.5 |
| Range | | <0.0002 -- <0.0002 | <0.0002 -- <0.0002 | <0.5 - <0.5 |

**Table 1-A**

## Conclusion

The analyses specified in the consent order P-12-0450 have been completed for representative samples of the initially isolated formulations of the PMN substance manufactured between August 11, 2015 and August 10, 2016. The Company is required to annually analyze representative samples of the initially isolated formulation(s) and report these results to EPA, in a cycle complementary to the

This report fulfills this requirement.